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Standard Test Method for Concentration Limits of Flammability of Chemicals[1]

This standard is issued under the fixed designation E 681; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the lower and upper concentration limits of flammability of chemicals having sufficient vapor pressure to form flammable mixtures in air at one atmosphere pressure at the test temperature. This method may be used to determine these limits in the presence of inert dilution gases. No oxidant stronger than air should be used.

NOTE 1—The lower flammability limit and upper flammability limit are also referred to as the lower explosive limit (LEL) and upper explosive limit (UEL) respectively.

1.2 This test method is limited to an initial pressure of 101 kPa (1 atm) or less with a practical lower pressure limit of approximately 13.3 kPa (100 mm Hg). The maximum practical operating temperature of this equipment is approximately 150°C (302°F).

1.3 This test method is one of several being developed by Committee E-27 for determining the flammability of chemicals.

1.4 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.*

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precaution statements are given in Section 7.

2. Summary of Method

2.1 A uniform mixture of a gas or vapor with air is ignited in a closed vessel and the upward and outward propagation of the flame away from the ignition source is noted by visual observation. The concentration of the flammable component is varied between trials until the composition which will just sustain propagation of the flame is determined.

3. Definitions

3.1 *lower limit of flammability or lower flammable limit* (LFL)—the minimum concentration of a combustible substance that is capable of propagating a flame through a homogeneous mixture of the combustible and a gaseous oxidizer under the specified conditions of test.

3.2 *upper limit of flammability or upper flammable limit* (UFL)—the maximum concentration of a combustible substance that is capable of propagating a flame through a homogeneous mixture of the combustible and a gaseous oxidizer under the specified conditions of test.

3.3 *propagation of flame*—as used in *this method*, the upward and outward movement of the flame front from the ignition source to the vessel walls, which is determined by visual observation.

4. Significance and Use

4.1 The lower and upper limits of flammability of gases and vapors define the range of flammable concentrations in air.

4.2 Limits of flammability may be used to determine guidelines for the safe handling of volatile chemicals. They are used particularly in assessing ventilation requirements for the handling of gases and vapors.

NOTE 2—The break point between nonflammability and flammability occurs over a narrow concentration range at the lower flammable limit but is less distinct at the upper limit.

5. Interferences

5.1 This method is not applicable to certain readily oxidized chemicals. If significant oxidation takes place when the vapors are mixed with air, unreliable results are obtained. Flow systems designed to minimize hold-up time may be required for such materials.

5.2 Measured flammable limits are influenced by flame-quenching effects of the test vessel walls. The test vessel employed in this method is of sufficient size to eliminate the effects of flame quenching for most materials. However, there may be quenching effects, particularly on tests run at subambient pressures. For certain amines, halogenated materials, etc., which have large ignition-quenching distances, tests should be conducted in larger diameter vessels.

6. Apparatus

6.1 Figure 1 is a schematic diagram of the apparatus; details and dimensions are presented in Annex A1. The apparatus consists of a glass test vessel (*1*), an insulated chamber (*2*) equipped with a source of controlled-temperature air (*3*), an ignition device with an appropriate power supply (*4*), a magnetic stirrer (*5*), and a cover (*6*)

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[1] This test method is under the jurisdiction of ASTM Committee E-27 on Hazard Potential of Chemicals and is the direct responsibility of Subcommittee E27.04 on Flammability and Ignitability of Liquid Chemicals.

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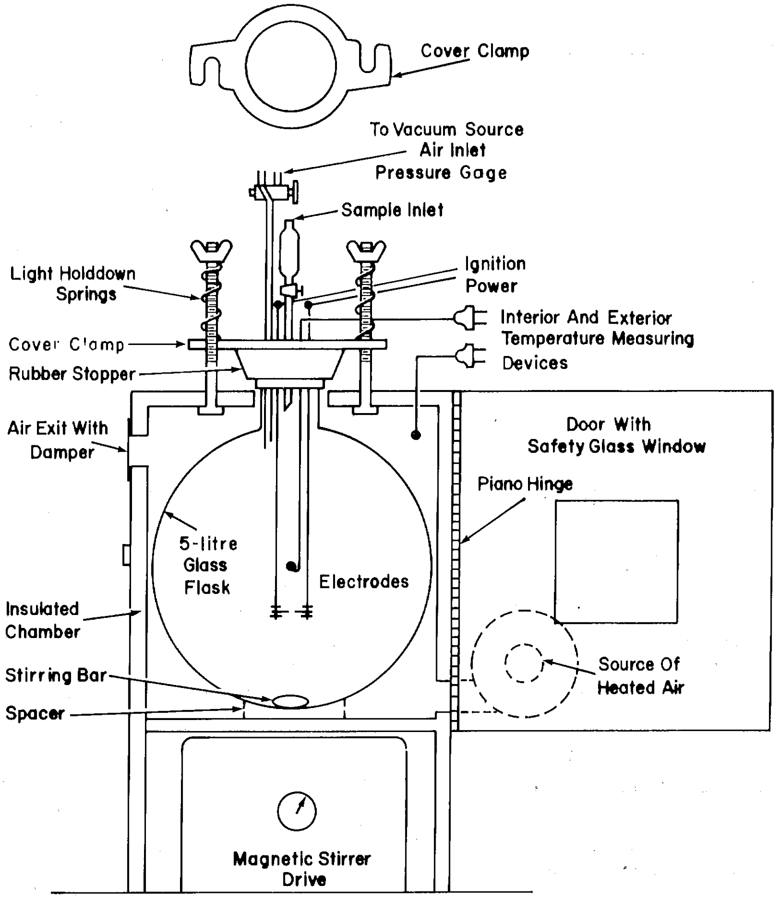

FIG. 1 **Schematic Diagram of Test Apparatus**

equipped with the necessary operating connections and components.

7. Safety Precautions

7.1 Tests should not be conducted in this apparatus with oxidizers stronger than air since explosive violence increases as oxidizer strength increases. Do not use oxygen, nitrous oxide, nitrogen dioxide, chlorine, etc., in this glass apparatus.

7.2 Adequate shielding must be provided to prevent injury in the event of equipment rupture, due to both implosions and explosions. A metal enclosure such as that recommended in A1.2 is one method suitable for this purpose.

7.2.1 Implosion of the test vessel at high vacuum levels is possible and, therefore, all evacuations must be made with the required shielding to protect against flying fragments.

7.2.2 Energetic explosions may be produced if tests are made at concentrations within the flammable range, between the LFL and UFL. The glass test vessel, equipped with a lightly held or loose cover, vents most explosions adequately. Nevertheless, shielding is required to protect against any probability of test vessel rupture. Methods for estimating initial test concentrations, discussed in Annexes A2, A3, and A4 may be employed to ensure that initial trials are conducted at concentrations less than the LFL or greater than the UFL.

7.2.3 In rare instances, particularly with upper limit tests, self-ignition may be encountered when air is rapidly introduced into the partially evacuated test vessel containing vaporized sample. Valves permitting remote operation, changes in sample and air introduction sequences, simple shields, and other techniques may be employed to ensure safe operations.

7.2.4 The test area should be equipped with electrical interlocks to prevent activation of the ignition source unless adequate shielding is in place.

7.3 Tests should not be conducted on thermally unstable materials which might undergo explosive decomposition reactions.

7.4 Tests should be conducted in a fume hood or other ventilated area to prevent personnel exposure to toxic chemicals or combustion products.

7.5 Precautions must be taken to ensure that the high-voltage spark ignition source does not contact temperature or pressure-measuring devices or other conductive paths which could create an electrical hazard to personnel or instrumentation outside the shielded area. Careful attention to electrical insulation integrity can reduce the possibility of hazard. Disconnects for all instrumentation lines will provide positive protection.

8. Equipment Calibration

8.1 Accurate determination of the flask volume is necessary for the calculation of flammable limits when sample measurement is on a weight or volume basis.

8.1.1 Determine the total volume of the flask as follows: Weigh a clean, dry flask with all components installed. Fill the flask with distilled water. Reinsert the cover, allowing the excess water to overflow, dry the outside of the flask, and reweigh. Record the difference in grams as the net volume of the flask in cubic centimetres. (Slight errors associated with water density differences are beyond the accuracy of this method.)

8.2 Calibrate pressure-, temperature-, and liquid-measuring devices against adequate standards.

9. Procedure

9.1 Assemble the equipment, as in Fig. 1, within an appropriate fume hood or other ventilated area and secure the door of the metal enclosure. The test vessel and all components should be clean and dry. Evacuate the system and flush with air to ensure removal of residual volatile materials that may be present as a result of cleaning or prior tests. As many as three evacuation/flush cycles may be required to ensure complete removal of combustion products between tests.

9.2 Adjust the flask to the desired test temperature. This temperature must be above the vapor condensation temperature of the mixture being tested.

9.2.1 It may be necessary to heat or insulate cover components and feed lines separately to prevent vapor condensation.

9.3 Record the actual barometric pressure at the test location.

9.4 Double check to make certain all safety precautions have been taken.

9.5 *Procedure for Sample Introduction as a Liquid:*

9.5.1 Evacuate the flask to a pressure of 1.33 to 6.65 kPa (10 to 50 mm Hg).

9.5.2 Place the desired liquid volume in a hypodermic syringe of appropriate size. Liquid volumes for initial trials may be estimated by methods given in Annex A2. Transfer the liquid to the inlet separatory funnel (see 9.5.4.1).

9.5.3 Turn on the stirrer at a minimum speed of 400 rpm.

9.5.4 Open the inlet stopcock. Allow the sample to be drawn into the flask. Close the stopcock when all the liquid has entered. Place a cover on the inlet separatory funnel.

9.5.4.1 A serum-bottle septum may be used in place of the separatory funnel. In this case, inject the sample directly into the flask by piercing the septum with the hypodermic needle. It will be necessary to make a volume correction if a significant volume of liquid is drawn from the needle or uncalibrated portion of the syringe.

9.5.5 When sample vaporization is complete, remove the separatory funnel cover and open the stopcock, permitting air to enter the test vessel slowly through the separatory funnel (see 7.2.3). Entering air sweeps traces of residual sample into the flask.

9.5.6 Release the cover hold-down devices and close the hood door.

9.5.7 Continue stirring for at least 5 min to obtain complete mixing and attainment of thermal equilibrium. Final trials should be made at longer mixing times to ensure optimal mixing conditions are achieved.

9.5.8 Turn off the stirrer.

9.5.9 Record the test temperature, T.

9.5.10 Disconnect instrumentation lines as required.

9.5.11 Darken the viewing area. Activate the ignition source. Observe for ignition and flame propagation away from the ignition source. Record as flammable any mixture producing a flame front that spreads from the ignition source and reaches the vessel walls.

NOTE 3—Mixtures having a composition just outside the flammable range exhibit a small cap of flame above the arc position; in some cases a vertical streak of flame may propagate to the vessel cover. (Absence of a cap flame may be an indication of insufficient ignition energy.) The onset of spherical, upward, and partial outward flame propagation signifies a limit or near-limit mixture. It is suggested that detailed observations of flame behavior be recorded on all trials. Include such notes as flame cap, no flame cap, upward and outward propagation, downward propagation, etc. These observations can serve as a guide to narrowing the region of uncertainty between go and no-go trials.

9.5.12 Vary sample size as required to find the minimum sample size, L_1, that gives flame propagation and the maximum sample size, L_2, below L_1, that does not give flame propagation. (The difference between L_1 and L_2 is a measure of the variability of the procedure for the material being studied.)

9.5.13 If numerous trials are required for a given series of tests, it may be necessary to remove the vessel for cleaning periodically, particularly for upper limit studies.

9.5.14 Final trials shall be made in a clean vessel.

NOTE 4—Ignition failures and inconsistent performance are occasionally encountered when highly conductive or very high ignition energy materials are tested using a spark ignition source. Limits for these materials should be determined using a fuse wire ignition source. Fuse wire ignition should also be used to confirm reduced pressure limit values arrived at on the basis of spark ignition source trials.

9.5.15 Record the values of the sample volumes L_1 and L_2. If partial propagation occurs over a range of sample sizes greater than 10 % of the sample size, the range should be specified in the report, for example, LFL = 5.4 ± 0.6 %).

9.5.16 Commence upper limit tests at a concentration greater than U_2, as defined in 9.5.17. Sample sizes for initial trials may be determined by methods given in Annex A3.

9.5.17 Record the values for the greatest sample quantity, U_1, that will propagate a flame and the least quantity, U_2, above U_1 that will not propagate a flame.

9.6 *Procedure for Sample Introduction as a Vapor:*

9.6.1 Sample concentration can be measured for gases and readily vaporized liquids on the basis of vapor pressure. In this instance, equip the vessel with a pressure transducer capable of reading to the nearest 0.0667 kPa (0.5 mm Hg). The system must also be capable of maintaining a vacuum of 0.0667 kPa or less.

9.6.2 Evacuate vessel and sample lines to a pressure of 0.0667 kPa (0.5 mm Hg or less).

9.6.3 Introduce the sample as a vapor through an appropriate inlet valve until the desired pressure is achieved. Introduce air as in 9.5.5, raising the pressure to atmospheric.

9.6.4 Carry out steps 9.5.6 through 9.5.17 as needed.

9.7 *Procedure for Sample Introduction as a Solid:*

9.7.1 Chemicals having melting points above room temperature but which totally melt and vaporize or totally sublime at the test conditions may be added to the test vessel as solids.

9.7.2 Bring the test vessel to atmospheric pressure (prior evacuation must be employed, as in 9.1, to ensure cleanliness).

9.7.3 Place the desired sample weight in the flask by raising the cover and inserting the sample.

9.7.4 Carry out steps 9.5.6 through 9.5.17 as needed.

NOTE 5—A small portion of the sample may be lost from the test vessel as the sample vaporizes and warms up to the test temperature. Losses are minimized by delaying the start of stirring until vaporization is complete. Maximum theoretical sample loss, which is small, may be readily calculated.

10. Calculations

10.1 Calculate the sample quantity, L or U, as follows:

$$L = \tfrac{1}{2}(L_1 + L_2) \qquad (1)$$

$$U = \tfrac{1}{2}(U_1 + U_2) \qquad (2)$$

where:

L = sample quantity used to calculate the lower flammable limit by Eq 3, and

U = sample quantity used to calculate the upper flammable limit by Eq 3.

For L_1 and L_2, see 9.5.12 . For U_1 and U_2, see 9.5.17.

10.2 Calculate the lower (LFL) and upper (UFL) flammable limits from the sample quantities. Ideal vapor phase behavior is assumed. (See Appendix X5.2 for a sample calculation and X5.1 for development of Eq 3.)

10.2.1 *Liquid Samples* (ideal vapor phase behavior is assumed):

$$\text{LFL} = \frac{(L_v)(d)(T)}{(\text{MW})(P)} \times \frac{(22.4)(P_o)(100)}{(V)(T_o)} \qquad (3)$$

where:

LFL = lower flammable limit in mole or volume %,

L_v = L = sample volume from Eq 1, cm^3,

d = sample density, g/cm^3,

T = test temperature, K,

MW = sample molecular weight, g, and

P = test pressure, absolute, kPa (mm Hg).

10.2.1.1 The second term is a constant for a given test apparatus where: P_o = standard pressure (760 mm Hg), V = vessel volume (litres), and T_o = standard temperature (273 K). (Any set of consistent units may be used for these calculations.)

10.2.1.2 Calculate upper flammable limits by replacing LFL with UFL and L_v with U_v in Eq 3.

10.2.2 *Vapor Samples* (ideal vapor phase behavior is assumed):

$$\text{LFL} = (L_p/P) \times 100 \qquad (4)$$

where:

L_p = L = sample partial pressure (mm Hg) from Eq 1.

10.2.2.1 Calculate upper limits by replacing LFL with UFL and L_v with U_v.

10.2.3 *Solid Samples* (ideal vapor phase behavior is assumed):

10.2.3.1 Calculate lower flammable limits by using Eq 3 with the terms (L_v) (d) replaced by L_w where: L_w = L = sample weight (g) from Eq 1.

10.2.3.2 Calculate upper flammable limits by replacing LFL with UFL and L_w with U_w.

10.3 *Complex Liquids, Solids, and Mixtures*—Flammability limits of some materials cannot be calculated in terms of mole or volume percent (Eq 3) since the molecular weight of the vapors is not known. This occurs in the case of unknown materials, multicomponent mixtures and materials exhibiting nonideal vapor phase behavior. It is more meaningful to express these limits in terms of weight of combustible per unit volume of mixture (mg/litre).

NOTE 5—Such limits are often given in the literature[2] as weight of combustible per litre of air at standard conditions (0°C and 760 mm Hg). These limits may be calculated from the following expression or by a similar expression for UFL:

$$\text{LFL, mg/litre} = \frac{\text{LFL(volume \%)}}{[100 - \text{LFL(volume \%)}]\left[\dfrac{22.414(\text{litre})}{\text{MW (mg)}}\right]}$$

10.3.1 Calculate lower flammable limits of mixed vapors and materials exhibiting nonideal vapor phase behavior as follows:

$$\text{LFL}_w = \frac{L_w}{V} \quad \text{or} \quad \frac{L_v(d)}{V} \times 100 \qquad (5)$$

where: LFL_w = lower flammable limit, in milligrams per litre.

10.3.2 Calculate upper flammable limits using Eq 5 replacing LFL_w with UFL_w, L_w with U_w, and L_v with U_v.

11. Report

11.1 Report flammability limits, LFL and UFL, calculated in accordance with Eqs 3, 4 or 5, along with the test temperature, test pressure, and ignition source (spark or fuse wire) used.

11.2 Report the limits initially in accordance with the units of measurement used in the determinations, that is, on a volumetric basis (mole or volume percent) for gases or vapor samples and on a gravimetric basis (milligrams per litre) for liquid or solid samples.

11.3 By substitution in Eq 3, calculated limits may then also be given for the gases or vapors on a gravimetric basis and for the liquids or solids on a volumetric basis, provided molecular weights of the combustibles are known. The report shall note if nonideal vapor phase behavior is suspected or known to occur.

11.4 Report the test variability if it exceeds 10 % of the sample size (see 9.5.1.5).

12. Precision and Bias

12.1 The precision and bias of this method have not yet been established.

12.2 The minimum precision of the method shall be ±0.5 % for limits of 10 volume %. Test data available at present are inadequate to establish any measure of repeatability or reproducibility.

ANNEXES

(Mandatory Information)

A1. DIMENSIONS AND SPECIFICATIONS OF APPARATUS (Fig. 1)

A1.1 *Test Vessel*—The test vessel shall be a borosilicate glass boiling flask, short-ring neck, 5000-cm^3 capacity, approximately 222 mm (8¾ in.) in diameter and 305 mm (12 in.) in height.

A1.2 *Insulated Chamber*—The dimensions shall be as follows:

Inside: 279 by 279 by 305 mm (11 by 11 by 12 in.) high
Height: 483 mm (19 in.). Adjust to accommodate stirrer unit.
Rear panel: ≥200 by 200-mm vent area
Top hole: 70.0-mm (2¾-in.) diameter
Air inlet hole: to fit air supply unit
Air exit hole: to accommodate a simple slide damper

A1.2.1 *Materials*—Sheet metal of at least 16 gage covered with insulation. Generally a portion of the metal bottom must be partially removed and replaced with nonmagnetic material to permit operation of the magnetic stirrer. The rear panel should be equipped with a vent (≥ 200 by 200 mm) providing explosion relief at low over-pressures, ≤6.9 kPa (1 psi). A lightly held panel of insulating board may be used.

A1.2.2 *Door,* hinged and latched, fitted with a 102 to 127-mm (4- to 5-in.) square safe viewing window made of polycarbonate and at least 12.7-mm (½-in.) thickness or equivalent.

A1.2.3 *Bolts,* top-fitted with two ¼-20 bolts on 127-mm (5-in.) centers to secure test vessel cover.

A1.2.4 *Spacer*—A cylindrical spacer constructed of perforated metal and sized to position the top of the neck of the test vessel just above the top of the test chamber permits air circulation and facilitates insertion and removal of the test vessel.

NOTE A1.1—If heavy construction is employed for the front, top, and side walls of the chamber and in the front of the base area, and if the rear and bottom panels of the chamber are of lightweight materials, explosion venting will be to the rear, away from the operator, in the event of vessel rupture.

A1.2.5 *Alternatives*—Other thermostated chambers or ovens and heating means may be employed if they permit temperature control and proper test manipulation and observation with adequate safety.

A1.3 *Heater*—Heated air is supplied from a blower, at the rate of approximately 0.38 m^3/min (13.5 ft^3/min), feeding air through a variable electric heater of approximately 2400 W. Commercial blowers, heaters, and manual or automatic controls and combinations thereof are available.

A1.4 *Ignition Device:*

A1.4.1 *Electrode Rods,* 3.175 to 4.76-mm (⅛ to 3⁄16-in.) diameter stainless steel, 317.5 mm (12½ in.) long. The upper ends are threaded for connection to a high-voltage source and the lower ends are threaded for attachment of spark gap points, or fuse wire, or both. Electrode rods are threaded for attachment of spark gap points, or fuse wire, or both. Electrode rods are spaced at least 32 mm (1¼ in.) apart. Other materials of construction may be used as needed.

A1.4.2 *Spark Gap,* having 6.4-mm (¼-in.) electrode spacing. Gap electrode extensions may be fabricated of platinum or tungsten wire held in wire connector lugs.

A1.4.3 *Fuse Wire*—19-mm (¾-in.) loop of 40-gage copper wire attached to threaded electrode rods in place of spark gaps.

A1.4.4 *Power,* approximately 30 mA at 15 kV, supplied by the secondary of a 120-V, 60-Hz luminous tube transformer, or by an equivalent device. Power for the fuse wire is 120 V, 60 Hz.

A1.4.5 *Timer,* to limit spark duration to 0.2 to 0.4 s. Commercial interval timers are available.

A1.5 *Stirring Devices:*

A1.5.1 *Stirring Bar,* 63.5-mm (2½-in.) egg-shaped, plastic-coated, magnet bar.

A1.5.2 *Drive*—Laboratory magnetic stirrer capable of functioning through the bottom of the test chamber and vessel.

A1.6 *Test Vessel Cover*—The cover can be constructed of a No. 14 rubber stopper with necessary holes for electrodes, sample inlet device, air inlet and evacuation connection and temperature-measuring device (see Fig. 1). *It is important to note* that the stopper rests on top and not inside the neck of the flask in order to facilitate venting.

NOTE A1.2—It is possible to operate at temperatures greater than 150°C (302°F) and to obtain more positive vacuum sealing through the use of specially constructed metal covers. High temperature O-ring seals for the flask top and inlet separatory funnel, and ceramic feedthroughs for the spark ignition source may be employed.

A1.7 *Cover Retainer* (Fig. 1)—This device held in place with wing nuts, light springs, and ¼-20 bolts can improve vacuum tightness of the test vessel when used to clamp down on the vessel cover.

A1.8 *Temperature Measurement:*

A1.8.1 Thermocouple, thermistor, resistance thermometer or other device may be used. High response speed is not required but is desirable in order to sense the heating effects associated with air introduction (7.2.3).

NOTE A1.3—Certain bare wire thermocouples may cause catalytic oxidation of test vapors, as evidenced by a persistent high-temperature excursion of the thermocouple junction. If this occurs, other thermocouple materials should be employed.

A1.8.2 A temperature-measuring device outside the test vessel but in the heating chamber or inlet air stream can aid in controlling test temperature.

A1.9 *Pressure Measurement*—Any pressure-measuring system accurate to 0.067 kPa (0.5 mm Hg) in the range from 0.067 to 101.3 kPa (0.5 to 760 mm Hg) and capable of being operated at temperatures greater than the condensation temperature of the materials under test is adequate for this unit. Active volume outside the test vessel itself should be held to a minimum since all components must be above the condensation temperature of the materials being tested. Electrical heating tapes may be employed for heating components to the desired temperature.

A2. ESTIMATION OF LOWER FLAMMABLE LIMITS FOR THE PURPOSE OF SELECTING SAMPLE SIZE FOR INITIAL TESTS

A2.1 It is the responsibility of the operator to ensure that adequate safety measures are employed in selecting sample sizes and in running the flammability limit tests. These guidelines are intended to assist the operator in planning the sequence of testing to avoid mixture compositions that may cause test vessel failure.

A2.2 To avoid energetic reactions, it is important that lower limit tests commence at a concentration below the lower flammable limit. Estimated lower flammable limits may be used in conjunction with Eq 3 to calculate a starting sample size.

A2.2.1 Several methods of estimating lower flammable limits are listed below. The accuracy of these methods varies, with some being relatively precise for certain classes of chemicals. Experience with these methods will improve the ability to make a reasonable evaluation as to the precision of the estimate with various types of material.

A2.3 Lower flammable limits of most organic chemicals are in the range from 40 to 60 mg/litre,[2] (see Note 5). For most materials, a sample size equivalent to 35 mg/litre may be used for initial tests. However, reactive fuels such as hydrogen and diborane have lower limits considerably below 30 mg/litre. As has been emphasized, lower limit tests should be conducted with extreme caution and upper limit tests should not be conducted in this apparatus on such materials (see 7.2).

A2.4 Lower flammable limits may be estimated from closed-cup flash point and vapor pressure data. The following approximate relationship may be employed:

$$\frac{P_f}{P_o} \times 100 = \mathrm{LFL_e} \qquad \text{(A1.1)}$$

where:

$\mathrm{LFL_e}$ = estimated lower flammable limit, volume %,

P_f = vapor pressure of combustible at the closed-cup flash point, mm Hg,

P_o = standard atmospheric pressure = 760 mm Hg.

A2.4.1 The validity of the flash point and vapor pressure data must be established in order to ensure a reasonable estimate of the lower flammable limit.

A2.4.2 The closed-cup flash point may not represent the lowest temperature at which a material evolves flammable vapor. Therefore, a safety factor must be employed when using this method.

A2.5 Lower flammable limits may be estimated as a function of the stoichiometric composition;[2,3] that composition where complete combustion to CO_2 and H_2O and conversion of the halogens, X, to HX consumes all the oxygen in the system.

A2.5.1 The stoichiometric composition may be calculated making use of balanced chemical equations for the combustion reaction.

A2.5.2 For combustion in air, this calculation reduces to the following equation:

$$C_s = \frac{100}{1 + 4.773\left[n + q + \frac{(m - k - 2p)}{4}\right]} \qquad \text{(A1.2)}$$

where:

C_s = stoichiometric composition of the combustible in air or mol %,

n = number of carbon atoms in the molecule,

m = number of hydrogen atoms in the molecule,

p = number of oxygen atoms in the molecule,

q = number of nitrogen atoms in the molecule, and

k = number of halogen atoms in the molecule.

A2.5.3 For saturated compounds containing only carbon, hydrogen, and oxygen, the lower flammable limit equals approximately 0.54 times the stoichiometric composition.[2,3]

$$\mathrm{LFL_e} = 0.54\, C_s \qquad \text{(A1.3)}$$

A2.5.4 For saturated compounds other than those containing carbon, hydrogen, and oxygen, lower flammable limits are generally greater than 0.54 times the stoichiometric composition.[2] Since data are limited and values vary, it is recommended that the factor 0.54 be used in calculating lower flammable limits from calculated stoichiometric composition for all saturated, nonreactive hydrocarbons.

A2.6 Lower flammable limits may also be estimated on the basis of correlations of known lower flammable limits of materials which are members of the same homologous series.

A2.7 Lower flammable limits of known mixtures may be estimated from known lower flammable limits of the mixture components using Le Chatelier's law. Details of these calculations can be found in *Bulletin 627*[2] and other references.

A3. ESTIMATION OF UPPER FLAMMABLE LIMITS FOR THE PURPOSE OF SELECTING SAMPLE SIZE FOR INITIAL TESTS

A3.1 Upper limits cannot be predicted as precisely as lower limits. Therefore, wide safety factors should be employed when using estimated upper limits to establish sample sizes for initial flammable limit tests (see Section 7).

A3.2 For saturated compounds containing only carbon, hydrogen, oxygen, and halogens, the upper flammable limit is generally greater than 3.5 times the stoichiometric composition.[3]

$$\mathrm{UFL_e} \geq 3.5\, C_s \qquad \text{(A1.4)}$$

where: $\mathrm{UFL_e}$ = estimated upper flammable limit.

NOTE A3.1—Equation A1.4 is not valid, and upper limits exceed 3.5

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[2] Zabetakis, M. G., "Flammability Characteristics of Combustible Gases and Vapors," *Bulletin 627*, U.S. Bureau of Mines, XMBUA, 1965.

[3] Hilado, C. J., "A Method for Estimating Limits of Flammability," *Journal of Fire and Flammability*, Vol 6, April 1965, p. 130.

times stoichiometric for certain unsaturated compounds, organic oxides, ethers, amines, and other reactive materials. It is suggested (see Section 7) that, with the exception of amines, upper limits not be conducted on such materials in this apparatus.

A3.2.1 The upper flammable limit estimated with Eq A1.4 may be used with Eq 3 to estimate a starting sample size for tests.

A4. CORRELATION OF KNOWN UPPER AND LOWER FLAMMABLE LIMITS WITH STOICHIOMETRIC COMPOSITION

A4.1 Details on a correlation of known upper and lower flammable limits with stoichiometric composition are described by Hilado.[3] These details may permit more exact estimation of lower and upper flammable limits than is proposed in A2 and A3. However, to quote from the reference, "These estimates must be very cautiously applied because there are many chemicals with unusual flammability characteristics. Estimated values are not a substitute for experimental data."

APPENDIX

(Nonmandatory Information)

XI. CALCULATION OF FLAMMABLE LIMITS FROM TEST DATA

XI.1 Basis of Eq 3

$$\text{LFL} = \frac{\text{moles of combustible in test vessel}}{\text{total moles of gas in test vessel}} \times 100 \quad \text{(X1)}$$

$$= \frac{(L_v)(d)}{\text{MW}} \times \frac{1}{\frac{V}{22.4} \times \frac{P}{(P_o)} \times \frac{(T_o)}{T}} \times 100 \quad \text{(X2)}$$

where:

LFL = lower flammable limit, mol or volume %,

L_v = L = liquid sample volume, cm^3, from Eq 1,

d = sample density, g/cm^3,

MW = sample molecular weight,

V = test vessel volume, litres,

22.4 = standard molar volume, litres,

P = test pressure, mm Hg,

T = test temperature, K,

P_o = standard pressure = 1 atm (760 mm Hg),

T_o = standard temperature = 273 K,

$$\text{LFL} = \frac{(L_v)(d)(T)}{(\text{MW})(P)} \times k \quad \text{(X3)}$$

$$k = \frac{(22.4)(P_o)(100)}{(V)(T_o)} \quad \text{(X4)}$$

X1.2 Sample Calculation

X1.2.1 Tests conducted in a 5100-cm^3 vessel at 24°C and 750 mm Hg with a liquid sample having a density of 0.775 g/cm^3 and a molecular weight of 84.16 produced the first flame propagation at 270 μl and no flame propagation at 260 μl.

L_v = ½ (L_1 + L_2) = ½(0.270 + 0.260) = 0.265 cm^3

d = 0.775 g/cm^3

MW = 84.16

V = 5.1 litres

P = 750 mm Hg

T = 24°C = 297 K

X1.2.2 Using Eq X4:

$$k = \frac{(22.4)(760)(100)}{(5.1)(273)} = 1222.7$$

This value will remain unchanged for all tests conducted in a system of 5.1-litre total volume.

X1.2.3 Using Eq 3 (see Section 10 on Eq X3):

$$\text{LFL} = \frac{(0.265)(0.775)(297)}{(84.16)(750)} \times 1222.7 = 1.18$$

where the lower flammable limit (LFL) = 1.18 volume % at 750 mm Hg pressure and 24°C.

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